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(71) Applicant: ELTECH SYSTEMS CORPORATION,
Corporate Headquaters 6100 Glades Road/Suite 305,
Boca Raton Florida 33434 (US)

(72) Inventor: Nguyen, Thinh, 20, Avenue du Gros Chêne,
Onex (CH)
Inventor: Wiaux, Jean-Pol, 239, rte. d'Annecy,
Croix-de-Rozon (CH)
Inventor: Vance, Christopher J., 46, Chemin de la
Montagne, Chêne-Bougeries (CH)

(74) Representative: Kügele, Bernhard et al, c/o DST SA 3,
Route de Troinex, CH-1227 Carouge/GE (CH)

(54) Coating for metallic substrates, method of production and use of the coating.

(57) A coating comprising aluminum and at least one of the transition metals of the groups IVB, VB and VIB are prepared comprising a unique microstructure which may be a solid solution of the transition metal in an aluminide matrix or a pseudo solid solution of the transition metal in an aluminum matrix. By means of special processes a super fine distribution of the transition metal within the matrix may be obtained in the form of a metastable pseudo solid solution even though thermodynamic considerations disfavour such fine distributions. The coating may, with advantage, be applied to superalloys and be heat treated together with the substrate in order to form a stable aluminide coating comprising transition metal dispersions in an aluminide matrix. One of the preferred manufacturing processes of such coatings is the electrodeposition thereof from non-aqueous electrolytes.

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COATING FOR METALLIC SUBSTRATES, METHOD OF PRODUCTION AND USE OF THE COATING

Field of Invention

The invention relates to a coating on a metallic substrate, the coating comprising an alloy of an aluminum-comprising component and at least a second component. The invention further relates to a method of producing this coating and finally the invention relates to the use of the coating.

Background of the Invention

It is known to apply on metallic substrates coatings of other metals or alloys in order to provide the substrates with superior properties due to the properties of the coating material itself or due to synergistic effects of the combination of substrate and coating.

Thus, it is known to provide metallic substrates with a coating of aluminum, which renders the coated substrate improved corrosion resistance due to the film-forming character of the aluminum. Such coatings may comprise pure aluminum or predeposited aluminum which was transformed

into an aluminide coating by interdiffusion of substrate components during a special heat treatment.

It is known that the addition of small quantities (lower than 1w%) of an "oxygen active element" such as Ti, Zr, Hf, Ta, Nb and others can improve the oxidation/ corrosion resistance of aluminide coatings by improving the adherence and the ductility of the Al_2O_3 scale to avoid its spalling during thermal shocks. Generally these additive elements are incorporated initially in the substrate materials, and diffuse into the coating layer during its formation. More often these elements must be stabilized in the substrate material, under the form of nitrides, carbides or stable intermetallic compounds, in order to avoid their massive diffusion to the coating zone, which would produce a detrimental effect on the coating performance (see Galmich, P. l'Aéronautique & l'Astronautique, no 8.1969/1 pages 31-39, or French Patent no. 2 207 198).

With respect to methods of producing such coatings, a variety of techniques are available for physical or chemical deposition of the coating materials on the substrates. However, it was found that a considerable number of these techniques yield unsatisactory coatings, e.g. due to the brittleness of the coating, bad adhesion, unsuitable surface conditions and other reasons.

One of the techniques for forming a coating comprising an alloy of aluminum and a second component uses electroplating methods in a non-aqueous electrolyte. As described in the US patent 3,775,260 the non-aqueous solvent may be e.g. toluene and the salts of the metals to be electroplated are added to the solvent in the form of bromides and/or chlorides.

However, this US patent does not disclose the electro-deposition of metals or alloys of Al with transition metals of the groups IVB, VB, and VIB of the periodic table or alloys thereof.

From the "Journal of Electrochemical Society", January 1957, page 21, it is known to electrodeposit Ti alloys from a nonaqueous bath, specially from ether containing Al complexes. Deposits of maximum 6% Ti, rest Al, could be obtained. Deposits with higher contents of Ti could not be achieved. Moreover, because of the high flammability of ether and its low electrical conductivity the practical operating conditions are highly disadvantageous. The amount of titanium or other transition metals as defined above in the coating according to the invention is advantageously far beyond the 6% level of the above disclosure, even though for certain combinations of alloy components or uses of the coating a lower level may be sufficient to obtain the desired properties. However, the mentioned article is quiet about aspects relating to the structure of the coating obtained by the disclosed process, nor does it propose to use such coatings for corrosion protection.

In the case of aluminide coatings a beneficial effect of transition metals on the adherence of the Al_2O_3 scale is observed but only if the transition metal concentration in the coating is lower than 1w%. It appears, however, that this concentration is too low to obtain any effect on the stability of aluminide coatings towards the interdiffusion interactions with the substrates.

By the conventional thermal diffusion processes, it is impossible to obtain a higher transition metal concentration in the coatings without any detrimental

effect on the oxidation resistance of aluminide coatings. In fact, due to the high operating temperature of the process and to the simultaneous outward diffusion of the principal component (Ni, Co, or Fe) and the transition metal of the substrate during the formation of the coating the structure of the latter is governed by thermodynamic factors of the constitutional diagram of the system. The transition metal content in the coating, under the form of a homogeneous solid solution is limited by its thermodynamic solubility in the aluminide phases, e.g. lower than 1w%. The excess of transition metal is segregated under the form of intermetallic compounds, the recrystallization of which under the form of large precipitates in the coating matrix is favoured by the high operating temperature and the slow cooling rate of the processes. Finally, the existence of transition metal-rich precipitates at the coating surface lead to catastrophic local oxidations due to the rapid diffusion rate of oxygen through the transition metal oxide scale (see "Coatings for High Temperature Applications, Ed. by E. Lang, Appl. Pub. p. 68).

Objects of the Invention

It is one object of the invention to provide an improved coating for a metallic substrate which has good corrosion protection properties.

It is an object of the invention to provide a coating, the structure of which is such that its stability towards interdiffusion with the substrate at high temperature is enhanced.

Another object of the invention is the provision of a coating which is applicable to complicated as well as simple structures.

It is a further object of the invention to provide methods of producing coatings on metallic substrates.

A still further object of the invention is to provide a method for electroplating the above mentioned metals or alloys in a maximum wide range of compositions of the deposit, whereby the desired composition of the deposit as well as the desired structure thereof may be obtained by suitable choice of additives and operating conditions such as plating current density, bath composition and method of bath preparation.

Finally it is an object of the invention to provide alternative methods for producing a coating as described above.

Summary of Invention

Coatings of the above mentioned type which have special properties in particular for corrosion protection are characterized in the second component being at least one transition metal of the groups IVB, VB or VIB of the periodic table, and the coating comprising a homogeneous distribution of the second component within the aluminum comprising component in form of a stable solid solution or a metastable, pseudo solid-solution of the transition metal or the compound thereof in the aluminum-comprising component.

Coatings according to the invention may advantageously be produced in a range of compositions from 2-95 w% of the transition metal or metals and 98-5 w% of aluminum. Of particular advantage are composition between 5-30 w% of titanium and 95 -70 w% of aluminum.

Coatings of the described structure and composition may be applied to any metallic substrate, but are specially advantageous in connection with so called super-alloys, comprising nickel, cobalt and iron, but also on other metals comprising valve metals which are difficult to electroplate because of their oxide surface layer, or other substrates comprising an intermediary layer comprising nickel.

The coatings as described above may be submitted to a preliminary or in situ heat treatment (during the beginning of its use) which may be carried out in order to form an interdiffusion layer which extends at least over a part of the depth of the coating. By the interdiffusion of the aluminum phase of the coating by e.g. nickel coming from the substrate a nickel aluminide coating is produced of extremely high corrosion resistance at high temperatures.

Such heat treated coatings may comprise a matrix of NiAl, CoAl, FeAl (or e.g. TiAl if the substrate contains Ti) wherein the transition metal is contained as a dispersed phase.

The final composition of such heat treated coatings may comprise 2-50w% of the transition metal(s) and 98-50w% of the aluminide phase.

In the case of Ti as the transition metal, the content thereof may be within 2-20w% and the aluminide phase 98-80w%.

It was found that the specific structure which provides very good corrosion resistance may advantageously be obtained by electroplating the coating onto the substrate in a non-aqueous electrolyte.

It is believed, without restriction to a certain theory, that the electroplating of both alloy components in non-aqueous electrolytes at low temperatures as it is in principal discribed in the European patent application 84810608.4 leads to a highly uniform distribution thereof, and due to the process conditions this distribution is maintained and "frozen" even though thermodynamic considerations disfavour such super fine distributions. In the case of a titanium-aluminum coating the structure comprises, thus, a metastable pseudo solid-solution, which expression defines a structure similar to a solid-solution with a distribution of the dissolved substance within the solvent on an atomic level. Compositions of the alloy components which fall within the theoretical solubilities (less than 1 w%) of the system form a stable solid solution.

One of the preferred methods to obtain the above comprises therefore electroplating at least one or an alloy of several transition metals of the groups IVB, VB and VIB of the periodic table of elements or an alloy of one or more of said transition metals with Al at near ambient temperature onto an electrically conductive substrate in a non-aqueous electrolyte, characterized in that the electroplating is carried out in an electrolyte comprising an aromatic hydrocarbon and an Al halide, wherein said

transition metal(s) is (are) dissolved in the form of halides of a high oxidation state and in that said transition metal(s) is (are) pre-reduced to a lower oxidation state.

The structure which may be obtained by one of the methods according to the invention is metastable, since the two components of the coating alone without the influence of the substrate material would segregate when heated up to a certain temperature. The definition of "pseudo" solid-solution relates to the fact, that the two alloyed metals would not form a solid solution by simple alloying techniques. However, according to one aspect of the invention, the coating may be heat treated prior to or during its use, whereby the metastable pseudo-solution is transformed into a stable solid solution of compounds of the coating components and components of the substrate which penetrate the coating by outwards diffusion during the heat treatment.

The coatings as described above may be used for corrosion protection in aqueous solutions of gaseous environments at temperatures up to 1400°C. As to their application for high temperature corrosion protection (up to 1400°C) a preliminary or in situ heat treatment may be carried out in order to form an interdiffusion layer which extends at least over a part of the depth of the coating.

<u>Detailed Description of the Invention</u>

In the literature the presence of Ti in an aluminide coating is reported to be detrimental to the coating due

to the growth of TiO_2 crystals which penetrate and crack the aluminum oxide surface. This effect is often observed when super alloys containing a high (3-5wt%) content of titanium are coated with aluminum, whereby outwards diffusion of the titanium creates the above problem.

It was, however, found that such detrimental effects do not occur when the titanium is dispersed within the aluminum matrix in a very fine distribution, a structure which is referred to hereinafter as metastable, pseudo solid-solution or a solid-solution proper, whereby the exact definition of this expression is a super fine distribution of the titanium within the aluminum which is similar to a solid solution. No real solid solution of titanium in aluminum is known, since the solubility of Ti in Al is below 1 w% at the considered temperature range. However, it is possible to produce such a distribution by special coating techniques, which are described hereinafter. The term "metastable" means that such fine, normally not existing distributions of Ti in Al represent some kind of "frozen" status, which would segregate if e.g. heated above the allotropic transformation temperature and rapidly cooled down. This transformation, however, does not take place if the Ti-Al system is present in the form of a coating on a substrate which contains components which interdiffuse with the coating during a suitable heat treatment, whereby a stable solid-solution of the titanium in a matrix of an intermetallic compound of aluminum and the respective substrate component is formed.

By outwards diffusion of components of the substrate such as e.g. nickel a ternary Ti-Al-Ni system is created which permits the stable super fine distribution.

It is not exactly known why the addition of relatively high amounts of titanium in the Al coating is beneficial, but it was demonstrated that the corrosion resistance of a coating according to the invention at both low and high temperatures is superior to a pure aluminum or an aluminide coating. In addition to the improved performance of such coatings by the very fine dispersion of Ti in the aluminum or aluminide matrix it was found that the presence of said dispersion limited the outwards diffusion of e.g. Ni from the substrate to an amount where only NiAl, a stable intermetallic compound, is formed and does not allow excessive outwards diffusion of the Ni, thereby inhibiting the formation of the Ni rich Ni_3Al intermetallic compound which is not specially useful for corrosion protection.

The super fine distribution of the titanium with the aluminum may be obtained by electroplating in a non-aqueous electrolyte.

For the use of such aluminum-transition metal coatings for high temperature corrosion protection it may be advantageous to submit the coating and the substrate to a heat treatment, which forms aluminide coatings by outward diffusion of components of the substrate.

Aluminide coatings according to the invention were formed by predeposition of an aluminum-transition metal alloy deposit which in certain cases was deposited on substrates comprising Ni, Co or Fe based alloys. In the latter cases, the predeposition step was followed by an interdiffusion reaction at elevated temperatures. This interdiffusion step may be carried out prior to the use of the coated substrate or during its use in situ.

Because of the good thermal stability of the pseudo solid solution predeposits, the diffusion reaction between the principal component of the substrate and aluminum of the deposit occurs under the solid form. During the formation step of the aluminide coating, the creation of local concentrations of transition metal was kinetically disfavoured by the low diffusion rate in solid phases, by the homogeneous composition of the initial deposits and by the higher solubility of Ni, Co or Fe in the aluminum matrix. After the formation of aluminide coatings the transition metal was surrounded by stable intermetallic compounds of NiAl, CoAl or FeAl, and the subsequent segregation of the transition metal dispersions in the coating matrix was thermodynamically unfavourable. Consequently the final coatings were composed of a matrix of NiAl, CoAl or FeAl intermetallic compounds, containing a uniform homogeneous and stable dispersion of transition metals, with a low concentration varying between 3 and 15% following the composition of the initial deposits. It was found that the presence of a uniform dispersion of transition metals in the aluminide coating in this concentration range improved greatly not only the adherence of the Al_2O_3 scale and the corrosion resistance towards sulphidisation but also the stability of the coating towards interdiffusion.

Because of the absence of the high local concentrations of transition metals in the coating matrix, no detrimental effect on the oxidation resistance of the coatings was observed.

Transition metal dispersed aluminide coatings provided by the invention were unique by their particular microstructure and were characterized by the following aspects:

- a formation reaction in the solid phase between aluminum from the predeposited coating and substrate components at 400-1200°C, in air or in an inert atmosphere;

- an aluminum source under the form of a pre-deposited layer of 30-50 micrometers of aluminum-transition metal pseudo solid solution containing 1-50w%, in particular 5-30w% of transition metals;

- a coating thickness obtained after diffusion of 50-80 micrometers composed of a matrix of NiAl, CoAl or FeAl intermetallic compounds containing a uniform homogeneous and stable dispersion of transition metal;

- a concentration of transition metals under the form of stable dispersion in the coating matrix in the range of 3-15 w%;

- a beneficial effect of the transition metal dispersion on the adherence of the Al_2O_3 scale, the corrosion resistance and the lifetime of the coatings.

In order to improve the performance of the transition metal dispersed aluminide coatings, a duplex coating and a modification of the chemical composition of the coating may be envisaged. It is known that the resistance of NiAl is better than that of CoAl or FeAl, and the performance of aluminide coatings produced by conventional/thermal diffusion processes is considerably improved by the presence of a pre-deposited layer of Ni.

Similar duplex coatings were applied to transition metal dispersed aluminide coatings. However, the method of preparation of aluminide coatings according to the invention allowed to obtain better results by optimizing the thickness of the Ni underlayer. Effectively, it was found that on optimal thickness of the Ni under-layer allowed to:

- obtain the desired structure of NiAl:

- obtain a thin Ni rich layer at the coating-substrate interface playing the role of a Ni diffusion barrier;

- avoid the rapid degradation of the coating matrix, and the formation of Kirkendall porosities due to a too thick Ni underlayer.

The optimum thickness of the Ni under layer was determined as a function of the composition and the thickness of the top layer of the aluminum-transition metal alloy deposit. For the useful composition range of the latter, the best results were obtained with a thickness of the Ni under layer according to the following equation:

$$\frac{\text{Thickness of Ni-under layer}}{\text{Thickness of alloy top layer}} = 0.25 - 0.50$$

The Ni under layer could be obtained by usual deposition techniques such as ion plating, plasma spraying, electroless deposition, electrodeposition etc...

EXAMPLES

Example 1

In a glove box with an inert atmosphere of nitrogen, a plating bath was prepared with the following molar composition:

$TiBr_4:AlCl_3:LiCl:Toluene = 0.033:0.330:0.198:1.00$

The Ti (IV) species were reduced to Ti (II) by reaction with an excess of about 5g of Al particles, at 60-80°C during 24 hours.

The electrolyte was placed afterwards in a cylindrical glass electrolysis cell. Two plane Al anodes of dimensions 5.0 x 2.5 x 0.2 cms were used. The agitation was insured by a magnetic stirrer.

A sample of Ni base superalloy Inconel 738 of dimensions 3.5 x 6.0 x 0.5cms was sandblasted, degreased in hot acetone and dried. Afterwards, the sample was introduced into the electrolysis cell. Before the deposition step, the surface of the superalloy sample was cleaned by an anodisation step, in the same electrolyte, at 4 mA/cm^2 with a charge corresponding to 5 $Asec/cm^2$. Afterward the deposition of TiAl alloy was made with a pulse current with $i_p = 20mA/cm^2$ and on:off = 1:1 msec. After the passage of a charge of 160 $Asec/cm^2$, a deposit thickness of about 40 um of TiAl (20 w% Ti, 80w% Al) was obtained.

After washing with water and acetone to remove the traces of electrolyte, the coated Inconel 738 sample was introduced into a furnace heated at 1000°C, in air. The diffusion treatment lasted 24 hours.

After the thermal treatment, a coating layer of 55 - 60 um was observed under the microscope. The thickenss of the coating was limited by a diffusion zone constituted by a chromium rich layer, resulting from the preferential diffusion of Ni from the Inconel 738 substrate. The composition of the coating, as shown in Table 1 below, and the X-ray images of different components were obtained by SEM method.

Table 1

Component	Coating composition As deposit (w%)	After diffusion (w%)
Al	82.6	21.7
Ti	17.4	3.8
Cr	0	6.2
Ni	0	61.8
Co	0	6.5

After diffusion the composition and the X-ray images of different coating components showed that the coating layer was principally composed of a matrix of NiAl with some CoAl containing a homogenous dispersed phase of TiAl or NiTiAl compounds and some Cr rich precipitates forming a solid solution of the minor constituents in the Ni/Al matrix.

Example 2

A TiAl coated sample of Inconel 738 was prepared as in example 1. The TiAl deposit composition and thickness were in the range of 20% Ti - 80% Al and 35-40 um.

The diffusion formation step of the aluminide coating from the TiAl deposit was performed directly under the test conditions.

The oxidation resistance of the coating was tested under thermal cycling conditions in static air. The thermal cycle was defined as follows: 23.5 hours at 1000°C followed by 0.5 hours at room temperature.

The oxidation resistance, and the stability towards diffusion of the coating were demonstrated by the evolution of the specific weight modification and the microstructure of the sample. The results after 2500 hours of exposure are listed in Table 2.

Table 2

Sample	weight gain (mg/cm_2)	oxidation penetration (um)	(Al/Ni) atomic ratio of coating
TiAl coated IN 738	+0.25	None	0.76
Uncoated	-19.0	200-300	-

These results demonstrate the good oxidation resistance and adherence of the Al_2O_3 scale developed at the coating surface by action of Ti dispersed phase, and the excellent stability of the coatings towards the outward diffusion of Ni from the substrate.

Example 3

A TiAl coated sample of Nimonic 90 (dimensions 2.5x6.0x0.15 cms) was prepared as in example 1. The deposit thickness and composition were in the range of 35-40 um and 20% Ti-80% Al.

The coated sample was submitted directly to the hot corrosion conditions simulated by spraying on the sample surface a solution of 0.9 mole/l of Na_2SO_4 + 0.1 mole/l K_2SO_4, in such a way that the dried salt load was in the range of 1.0 to 1.5 mg/cm^2. The hot corrosion test conditions were as follows:

Temperature: 900^{o}C
Salt load: 1.0 - 1.5mg/cm^2 every 48 hours
Thermal cycle: 47 hours at 900^{o}C - 1 hour at room temperature.

The corrosion resistance of the coating was demonstrated by the evolution of the specific weight gain and the microstructure of the sample. The results after 360 hours of exposure are listed in Table 3.

Table 3

Sample	weight gain (mg/cm^2)	sulfur penetration (um)	coating thickness after test (um)
Al coated Nimonic 90	0	None	35
Uncoated Nimonic 90	-3.8	150-200	-

CLAIMS

1. A coating on a metallic substrate, the coating comprising an alloy of an aluminum-comprising component and at least a second component, characterized in the second component being at least one transition metal of the groups IVB, VB or VIB of the periodic table or a compound thereof, the coating comprising a homogeneous distribution of the second component within the aluminum comprising component in form of a stable solid solution or a metastable, pseudo solid-solution of the transition metal or the compound thereof in the aluminum-comprising component.

2. The coating of claim 1, characterized by a composition of 1-95 wt% of the transition metal(s) and 99-5 wt% of Al.

3. The coating of claim 2, characterized by a composition of 5-30w% of titanium and 95-70w% of aluminum.

4. The coating of claim 3, characterized in comprising components of the substrate being introduced into the coating by thermal interdiffusion, the interdiffused components being at least one of nickel, cobalt and iron.

5. The coating of claim 4, characterized by the substrate comprising an intermediate layer containing nickel, iron, cobalt and/or titanium.

6. The coating of one of the claims 1-5, characterized by the coating being modified by a heat treatment during which at least a part of the coating is transformed into an interdiffusion zone wherein the metastable, pseudo solid-solution is transformed into a stable solid solution comprising aluminum, titanium and one of nickel, iron and cobalt, the heat treatment being carried out preliminary to or during its initial use in-situ at temperatures which allow interdiffusion of the deposit and components of the substrate.

7. The coating of claim 6, characterized by comprising a matrix of at least one of NiAl, CoAl, TiAl and FeAl, containing a dispersed phase of the transition metal(s).

8. The coating of claim 7, characterized by comprising 2-50w% of the transition metal(s) and 98-50w% of the aluminide phase.

9. The coating of claim 7 or 8, characterized by comprising 2-20w% of titanium and 98-80w% of the aluminide phase.

10. A method of manufacturing the coating of one of the claims 1-9 by electrodepositing an alloy of aluminum and at least one transition metal at near ambient temperature onto an electrically conductive substrate in a non-aqueous electrolyte, characterized by the electroplating being carried out in an electrolyte comprising an aromatic hydrocarbon and an aluminum halide, wherein said transition metal(s) is (are) dissolved in the form of halides of a high oxidation state, said transition metal(s) being pre-reduced to a lower oxidation state.

11. The method of claim 10, characterized in the coating and the substrate being heated to a temperature between 400-1200°C for 1-100 hours, to permit a solid phase formation reaction between the aluminum of the predeposited coating and substrate components.

12. The method of claim 11, characterized in the heating being carried out in air or in inert atmosphere.

13. A use of the coating of one of the claims 1-9 for corrosion protection in aqueous solutions or gaseous environments at temperatures up to 1400°C.

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(71) Applicant: ELTECH SYSTEMS CORPORATION,
Corporate Headquaters 6100 Glades Road/Suite 305,
Boca Raton Florida 33434 (US)

(72) Inventor: Nguyen, Thinh, 20, Avenue du Gros Chêne,
Onex (CH)
Inventor: Wiaux, Jean-Pol, 239, rte. d'Annecy,
Croix-de-Rozon (CH)
Inventor: Vance, Christopher J., 46, Chemin de la
Montagne, Chêne-Bougeries (CH)

(74) Representative: Kügele, Bernhard et al, c/o DST SA 3,
Route de Troinex, CH-1227 Carouge/GE (CH)

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EUROPEAN SEARCH REPORT

Application number

EP 85 81 0521

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl 4)
A	EP-A-0 023 762 (NIHON MEDEL) ---		C 25 D 3/56 C 25 D 5/50
D,X P,E	EP-A-0 148 122 (ELTECH SYSTEMS) * Whole patent * ---	1-13	
A	CHEMICAL ABSTRACTS, vol. 77, no. 26, 25th December 1972, page 521, abstract no. 171827u, Columbus, Ohio, US; Ch.V. GERASIMOVICH et al.: "Electroplating aluminum on copper and steel", & CHEM. CHEM. TECHNOL. 1971, No. 13, 275-8 ---		
			TECHNICAL FIELDS SEARCHED (Int. Cl 4)
A	CHEMICAL ABSTRACTS, vol. 67, no. 14, 2nd October 1967, page 6581, abstract no. 70034w, Columbus, Ohio, US; A. LEVINSKAS et al.: "The phase diagram and electrolysis of the aluminum bromide-diethyl ether system", & ELEKTROKHIMIYA 3(6), 781-5(1967) -----		C 25 D 3/56

The present search report has been drawn up for all claims

Place of search	Date of completion of the search	Examiner
THE HAGUE	07-10-1987	VAN LEEUWEN R.H.

CATEGORY OF CITED DOCUMENTS

X : particularly relevant if taken alone
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E : earlier patent document, but published on, or after the filing date
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